



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 2587

Trace Elements in Soil Containing Lead from Paint (Nominal 3000 mg/kg Lead)

This Standard Reference Material (SRM) is intended for use in the evaluation of methods and for the calibration of apparatus used to determine lead and other trace elements in soil. SRM 2587 is composed of soil samples collected from a suburban garden known to have been contaminated by lead-based house paint. All values are reported as mass fractions [1], on a dry mass basis and are based on measurements using a sample mass of at least 200 mg. A unit consists of approximately 55 g of material with a particle size of <75 μm (200 mesh).

Certified Concentration Values: The certified values for four environmentally important elements are provided in Table 1. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST.

Reference Concentration Values: Reference values for 14 additional elements are provided in Table 2. Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision and may not include all sources of uncertainty.

Information Concentration Values: Information values are listed in Table 3. These values may be of interest to the SRM user; however, insufficient information is available to adequately assess the uncertainty associated with these values.

Expiration of Certification: The certification of this SRM is valid within the measurement uncertainties specified until **30 September 2008**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see Use section). However, the certification will be nullified if the SRM is contaminated or modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

Partial financial support for the development of this SRM was provided by the U.S. Environmental Protection Agency (EPA) under the direction of project manager B. Schumacher of the EPA National Exposure Research Laboratory, Las Vegas, NV.

The overall direction and coordination of the technical measurements leading to the certification of this SRM were performed by R.L. Watters, Jr. and G.C. Turk of the NIST Analytical Chemistry Division.

Statistical consultation was provided by L.M. Gill of the NIST Statistical Engineering Division.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by B.S. MacDonald.

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Certificate Issue Date: 3 March 1999

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NOTICE AND WARNING TO USERS

Use: To relate analytical determinations to the certified values on this Certificate of Analysis, a minimum sample mass of 200 mg should be used and the sample should be dried according to the Instructions for Drying. Sample preparation procedures should also be designed to effect complete dissolution in order to relate the determined value to the certified value. The SRM must be stored in its original bottle at temperatures less than 30 °C away from fumes and direct sunlight.

Instructions for Drying: Samples should be oven dried for 2 h at 105 °C. For the determination of volatile elements (arsenic and mercury), samples should be analyzed as received; separate samples should be dried according to these instructions to obtain a correction factor for moisture. Moisture corrections are then made to the measurement results before comparing them to the certified values. This alternative drying method may also be used for nonvolatile elements.

Certified Concentration Values and Uncertainties: Certified values and uncertainties are provided in Table 1. The certified values are based on results from two or more NIST independent analytical methods and additional results from methods used at the U.S. Geological Survey (USGS) (Table 4). All values are reported as mass fractions [1], on a dry basis (see Instructions for Drying), and are based on measurements using a sample mass of at least 200 mg.

Table 1. Certified Mass Fractions

Element	Mass Fraction mg/kg		
Arsenic	13.7	±	2.3
Cadmium	1.92	±	0.23
Chromium	92	±	11
Lead	3 242	±	57

The certified values are equally weighted means from the combination of results provided by NIST and USGS. The uncertainty is calculated as, $U=ku_c + B$. The quantity u_c is the combined standard uncertainty calculated according to the ISO Guide [2] and is intended to represent, at the level of one standard deviation, the combined effect of within-method variation and material inhomogeneity. The coverage factor, k , is determined from the Student- t distribution corresponding to the calculated effective degrees of freedom and 95 % level of confidence for each element. B is a bias adjustment for the difference between methods, which is the maximum difference between the certified value and method means [3].

Reference Concentration Values and Uncertainties: Reference values and uncertainties are given in Table 2. The reference value for Hg is derived from a single method performed at NIST. The reference values for the remaining elements are derived from two or more methods performed at NIST and/or USGS.

Table 2. Reference Mass Fractions

Element	Mass Fraction mg/kg			Element	Mass Fraction mg/kg		
Aluminum	58 600	±	1 700	Phosphorus	970	±	100
Barium	568	±	12	Potassium	15 830	±	550
Calcium	9 270	±	200	Silicon	331 300	±	2 600
Iron	28 130	±	250	Sodium	11 270	±	330
Magnesium	6 690	±	250	Strontium	126	±	19
Manganese	651	±	23	Titanium	3 920	±	650
Mercury	0.290	±	0.009	Zinc	335.8	±	7.6

The reference values are equally weighted means from the combination of results provided by NIST and USGS. The uncertainty is calculated as, $U=ku_c + B$. The quantity u_c is the combined standard uncertainty calculated according to the ISO Guide [2] and is intended to represent, at the level of one standard deviation, the combined effect of within-method variation and material inhomogeneity. The coverage factor, k , is determined from the

Student- t distribution corresponding to the calculated effective degrees of freedom and 95 % level of confidence for each element. B is a bias adjustment for the difference between methods, which is the maximum difference between

the certified value and method means [3].

Information Concentration Values: Information values are provided in Table 3. The values are either from a single method or are the mean of two methods.

Table 3. Information Mass Fractions

Element	Mass Fraction mg/kg	Element	Mass Fraction mg/kg
Beryllium	9.2	Nickel	36
Cerium	57	Niobium	14
Cobalt	14	Scandium	11
Copper	160	Thorium	7.5
Gallium	13	Vanadium	78
Lanthanum	29	Ytterbium	1.6
Lithium	32	Yttrium	15
Neodymium	25		

COLLECTION, PREPARATION, AND ANALYSIS

Collection: Soil material used in the preparation of SRM 2587 was collected from a suburban residence in the Hartford, CT area known to be contaminated by lead-based paint. The soil was collected within the top 25 cm after surficial organic material had been removed. Visual inspection of the collected soil revealed moderate quantities of partially decomposed organic matter as well as minor amounts of masonry debris. Uncontaminated soil from the Boston, MA area was collected for use as a diluent.

Preparation: The preparation of SRM 2587 was performed at the USGS laboratory in Denver, CO, under the direction of S.A. Wilson. The collected soils were dried at room temperature for 5 days, disaggregated, and sieved. The material with particle size under 2 mm was collected in 20 L containers and chemically analyzed. The information from this preliminary analysis was then used to combine and blend the sub-sets into a single set with a target lead concentration of 3000 mg/kg. The blended mixture was ground to < 75 μm (200 mesh) using a Hardinger¹ ball mill equipped with an air separator system and mixed for 20 hours using a cross-flow V-blender. The material was then split into 8 kg aliquots and sterilized using Co-60 irradiation. After sterilization the material was re-combined, blended for 3 h, and bottled.

Analysis: Certification analyses were performed in the NIST Analytical Chemistry Division and at USGS. The analytical methods used and the analysts are listed in Table 4.

Table 4. Methods Used for the Analysis of SRM 2587

Method	Lab	Analysts	Elements ^a
Electrothermal Atomic Absorption Spectrometry	NIST	B. Buehler, M.S. Epstein	As, Cr
Flow Injection – Cold Vapor Atomic Absorption Spectrometry	NIST	M.S. Epstein	Hg
Inductively Coupled Plasma Mass Spectrometry	NIST	L.L. Yu	As, Cd
Instrumental Neutron Activation Analysis	NIST	R. Zeisler	As, Cr
Isotope Dilution – Inductively Coupled Plasma Mass Spectrometry	NIST	E.S. Beary, K.E. Murphy	Cd, Pb
X-Ray Fluorescence Spectrometry with Fusion Sample Preparation, Calibrated with Fusions of Mixed Pure Element Compounds	NIST	P.A. Pella, A.F. Marlow, E. Ramirez (Guest Scientist from Centro Nacional de	Al, Ba, Ca, Cr , Fe, K, Mg, Mn, P, Pb , Si, Sr, Ti, Zn

¹ Certain commercial equipment, instruments, or materials are identified in this report to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

		Metrologia, Mexico)	
Inductively Coupled Plasma Atomic Emission Spectrometry	USGS	P.H. Briggs	Al, As , Ba, Be, Ca, Ce, Co, Cr , Cu, Fe, Ga, K, La, Li, Mg, Mn, Na, Nb, Nd, Ni, P, Pb , Sc, Sr, Th, Ti, V, Y, Yb, Zn
X-Ray Fluorescence Spectrometry with Fusion Sample Preparation, Calibrated with Fusions of Geological Reference Materials	USGS	S.A. Wilson, J.S. Mee, D.F. Siems	Al, Ca, Fe, K, Mg, Na, P, Si, Ti

^a Certified Elements shown in **Bold**.

User Experience with SRM 2587: In order to demonstrate user experience with SRM 2587, a number of laboratories analyzed this material, using a variety of dissolution and instrumental methods. For lead, this was done through the Environmental Lead Proficiency Analytical Testing Program (ELPAT), where SRM 2587 was included as an unknown for Round Robin number 12. Data for As, Cd, Cr, and Hg, were supplied by volunteer laboratories in a round robin exercise organized by NIST. The sample preparation methods include EPA-SW846-3050A, EPA-SW846-3051 [4] and others. As these methods may not effect complete sample dissolution, the results obtained using these methods tend to be lower than the certified values. The results are summarized in Table 5. These results were not used in calculating the certified values of SRM 2587.

Table 5. Results of Round Robin Exercise

Element	Mean mg/kg	Minimum mg/kg	Maximum mg/kg	Std. Deviation mg/kg	N
As	12.2	7.1	16.6	2.2	20
Cd ^a	1.8	1.0	2.5	0.4	17
Cr	34	21	53	8	22
Hg	0.25	0.07	0.33	0.06	19
Pb	3 091	2 590	3 479	228	40

^a The results reported for Cd from four laboratories were erroneously high and are not included in the summary statistics given here.

REFERENCES

- [1] Taylor, B.N., "Guide for the Use of the International System of Units (SI)," NIST Special Publication 811, 1995 Ed., (April 1995).
- [2] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland, (1993): see also Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington DC, (1994).
- [3] Schiller, S.B. and Eberhardt, K.R., "Combining Data from Independent Chemical Analysis Methods," *Spectrochimica Acta*, **46B**, pp. 1607-1613, (1991).
- [4] Federal Register 1-13-95 SW-846 update #2, Final.

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Telephone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet <http://ts.nist.gov/srm>.

